RECEIVED
CENTRAL FAX CENTER
JUN 2 5 2007

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2001-080989

(43)Date of publication of application : 27.03.2001

(51)Int.Cl. C30B 15/10
C30B 29/40
C30B 29/42
H01L 21/208

(21)Application number : 11-250244

(22)Date of filing : 03.09.1999

(71)Applicant : HITACHI CABLE LTD

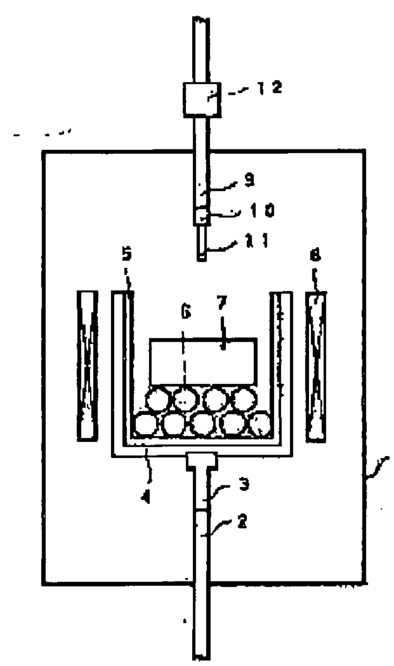
(72)Inventor : WACHI MICHINORI
YABUKI SHINJI
MIZUNIWA SEIJI
TAIHO KOJI
INADA TOMOKI

(54) DEVICE FOR PRODUCING COMPOUND SEMICONDUCTOR SINGLE CRYSTAL AND PRODUCTION PROCESS USING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To produce a long-size or large-diameter compound semiconductor single crystal with good reproducibility.

SOLUTION: This production process comprises: receiving a raw material melt and a liquid encapsulating agent in a heated crucible 5 that is received in a pressure vessel filled with an inert gas; and rotating a seed crystal 11 and the crucible 5 relatively (or reversely) to each other while bringing the seed crystal 11 into contact with the raw material melt, to produce a compound semiconductor single crystal by an LEC(liquid encapsulated Czochralski) method, wherein, as the crucible 5, a crucible having a diameter sufficient to provide the ratio of the diameter of the crucible to the diameter of such an aimed grown single crystal, i.e., (crucible diameter)/(crystal diameter) of 2.2-3.2, is used.



PAGE 17/30 * RCVD AT 6/25/2007 6:57:51 PM [Eastern Daylight Time] * SVR:USPTO-EFXRF-2/20 * DNIS:2738300 * CSID:512 327 5452 * DURATION (mm-ss):15-32

LEGAL STATUS

[Date of request for examination]	14.06.2002
[Date of sending the examiner's decision of rejection]	14.06.2005
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	
[Date of registration]	
[Number of appeal against examiner's decision of rejection]	
[Date of requesting appeal against examiner's decision of rejection]	
[Date of extinction of right]	

* NOTICES *

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] To the crucible which was held in the proof-pressure container filled up with inert gas, and was heated, raw material melt, In the manufacturing installation of the compound semiconductor single crystal by the LEC method into which seed crystal and a crucible are rotated relatively and a single crystal is grown up, containing liquid encapsulant and contacting seed crystal to raw material melt The manufacturing installation of the compound semiconductor single crystal characterized by setting the ratio {(crucible diameter) /(crystal diameter)} of the diameter of a crystal and a crucible to grow up to the range of 2.2-3.2.
[Claim 2] The manufacturing installation of the compound semiconductor single crystal characterized by the diameter of the crystal to grow up being a diameter crystal of macrostomia 100mm or more in the manufacturing installation of the compound semiconductor single crystal of claim 1.
[Claim 3] To the crucible which was held in the proof-pressure container filled up with inert gas, and was heated, raw material melt, In the manufacture approach of the compound semiconductor single crystal by the LEC method into which seed crystal and a crucible are rotated relatively and a single crystal is grown up, containing liquid encapsulant and contacting seed crystal to raw material melt The manufacture approach of the compound semiconductor single crystal characterized by growing up a compound semiconductor single crystal using the crucible which set the ratio {(crucible diameter) /(crystal diameter)} of the diameter of a crystal and a crucible to grow up to the range of 2.2-3.2.
[Claim 4] The manufacture approach of the compound semiconductor single crystal characterized by a diameter growing up the crystal of the diameter of macrostomia 100mm or more in the manufacture approach of the compound semiconductor single crystal of claim 2.

[Translation done.]

* NOTICES *

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the manufacture approach which used the manufacturing installation of a compound semiconductor single crystal, and it.
[0002]
[Description of the Prior Art] An III-V group compound semiconductor has come to be widely used for the electronic device of a high-speed integrated circuit, an opto-electronic integrated circuit, or others by quality improvement of the single crystal. inside -- gallium arsenide (GaAs) -- electron mobility -- silicon -- comparing -- early and 107 the wafer of the specific resistance more than omega-cm -- manufacture -- there is the description of being easy. In current, the above-mentioned single crystal of the half insulation GaAs is mainly manufactured by the liquid closure Czochralski method (LEC method).
[0003] The outline of the crystal-pulling equipment by the LEC method currently widely used for drawing 1 as a manufacturing installation of a GaAs single crystal is shown.
[0004] In drawing, 1 is an elevated-temperature furnace for crystal growth (proof-pressure container), into the elevated-temperature furnace 1, a lower shaft 2 is inserted from the bottom, and the susceptor 4 is supported through the pedestal 3 at the tip of this lower shaft 2. The crucible 5 made from PBN is arranged in the susceptor 4. The heater 8 is formed in the perimeter of a susceptor 4, and the crucible 5 made from PBN can be heated now from a perimeter through a susceptor 4. It connects with the rolling mechanism which is not illustrated and a lower shaft 2 is rotated with a fixed rotational speed. Moreover, from the elevated-temperature furnace 1 bottom, an arm shaft horizontal 9 is inserted in same axle with a lower shaft 2, and the seed crystal 11 ((100) is usually used as bearing) which had desired bearing in the seed crystal holder 10 formed in the lower limit is attached. Besides, while axial rotation is carried out in the crucible 5 made from PBN by the rotation and the elevator style which is not illustrated at the reverse sense, rise-and-fall migration of the shaft 9 is carried out. The weight sensor 12 is formed in the middle of the arm shaft horizontal 9, and the crystal weight of a growth process can be detected now by this.
[0005] In the case of crystal growth, it is Ga, As6 (Ga7470g, As8124g), and B-2 O3 in the crucible 5 made from PBN first. 4,000g of liquid encapsulants 7 is put in, evacuation of the inside of the elevated-temperature furnace 1 is carried out, 40 atmospheric-pressure extent is pressurized with inert gas, such as nitrogen or an argon, after that, it energizes at the main heater 8, and the temperature up of the interior of the crucible 5 made from PBN is carried out. The liquid encapsulant (B-2 O3) 7 becomes soft and dissolves around 500 degrees C, and it is a wrap about Ga and As6. A temperature up is carried out succeedingly, temperature of the crucible 5 made from PBN interior is made into 1,238 degrees C or more, GaAs is compounded, and it is made to dissolve further. Next, after decompressing the inside of the elevated-temperature furnace 1 in five to 20 atmospheric pressure, seed crystal 11 is dropped, the tip is dipped in raw material melt, and seed attachment is performed. Then, the arm shaft horizontal 9 is pulled up at the rate of 7 - 12 mm/hr, lowering the temperature of a heater 8, detecting crystal weight by the weight sensor 12, the output of a heater 8 is controlled and a GaAs single crystal is grown

PAGE 20/30 * RCVD AT 6/25/2007 6:57:51 PM [Eastern Daylight Time] * SVR:USPTO-EFXRF-2/20 * DNIS:2738300 * CSID:512 327 5452 * DURATION (mm-ss):15-32

up.

[0006] The product made from PBN (Pyrolitic Boron Nitride : pyrolysis boron nitride) is used for the crucible 5 used by the above-mentioned LEC method, and its crucible made from PBN is very expensive. As for the diameter of the crucible used by the LEC method from this, it is common to use a bigger thing a little than the diameter of the crystal to grow up.

[0007]

[Problem(s) to be Solved by the Invention] However, it is very difficult and the techniques of growing up a compound semiconductor single crystal are members (called a hot zone.), such as a heater of a heating means, and a thermal shield cylinder. It is difficult to acquire the growth conditions of a compound semiconductor single crystal with sufficient repeatability according to the arrangement described as Following HZ, a configuration, the quality of the material, etc., in order to be influenced more finely.

[0008] When it was going to obtain the diameter crystal of macrostomia for which especially the diameter of a compound semiconductor single crystal exceeds 100mm, it was very difficult to acquire the growth conditions of a compound semiconductor single crystal especially with sufficient repeatability, and it was rare that polycrystal-ized in the process of single crystal growth in almost all cases, and a single crystal all over the districts was obtained from the seed attachment section (seed section) of a crystal to the crystal growth last section (tail part).

[0009] Although not illustrated by drawing 1 , members (hot zone HZ), such as a thermal shield cylinder, are prepared in the elevated-temperature furnace 1, and it was regarded as the factor in which this arrangement of HZ, a configuration, the quality of the material, etc. influence the repeatability of the growth conditions of a compound semiconductor single crystal till recently. However, the factor is not only in HZ but it is becoming clear that the property of the crucible made from PBN to be used is also greatly related as stabilization of conditions, such as arrangement of HZ, a configuration, and the quality of the material, is attained. The crucible made from PBN does not react with the raw material compound at the time of the elevated temperature which grows a compound semiconductor single crystal, but is Pyrolitic Boron Nitride. With an advantage, like the purity of the very thing is high, it is an instrument especially indispensable to growth of a GaAs single crystal, and, for the moment, the substitute is not known. Therefore, when improving the repeatability of the growth conditions of a compound semiconductor single crystal and aiming at improvement in the yield, the property improvement of the crucible made from PBN is indispensable. However, in the property of the crucible made from PBN, it was not clear what was the most decisive factor.

[0010] Then, the purpose of this invention cancels the trouble of the above mentioned conventional technique, and is to offer the manufacturing installation and the manufacture approach of a compound semiconductor single crystal that a compound semiconductor single crystal can be obtained with sufficient repeatability.

[0011]

[Means for Solving the Problem] this invention persons acquired the following knowledge, as a result of repeating research wholeheartedly about manufacture of the compound semiconductor single crystal by the LEC method. That is, although it had only been thought that the factor for acquiring the growth conditions of a compound semiconductor single crystal with sufficient repeatability till recently required arrangement of HZ, a configuration, the quality of the material, etc. for HZ, it discovered that the factor for obtaining a compound semiconductor single crystal with sufficient repeatability is not only in HZ, and there was a factor also in the diameter of the crucible made from PBN to be used as stabilization of conditions, such as arrangement of HZ, a configuration, and the quality of the material, was attained. And by specifying the ratio of the diameter of a crucible, it found out that a compound semiconductor single crystal could be obtained with sufficient repeatability, and this invention was reached.

[0012] Namely, the manufacturing installation of the compound semiconductor single crystal of this invention Hold in the proof-pressure container filled up with inert gas, and to the heated crucible Raw material melt, In the manufacturing installation of the compound semiconductor single crystal by the LEC method into which seed crystal and a crucible are rotated relatively and a single crystal is grown up, containing liquid encapsulant

PAGE 21/30 * RCVD AT 6/25/2007 6:57:51 PM [Eastern Daylight Time] * SVR:USPTO-EFXRF-2/20 * DNIS:2738300 * CSID:512 327 5452 * DURATION (mm-ss):15-32

and contacting seed crystal to raw material melt The ratio {(crucible diameter) /(crystal diameter)} of the diameter of a crystal and a crucible to grow up is set to the range of 2.2-3.2 (claim 1).
[0013] Moreover, the manufacture approach of the compound semiconductor single crystal of this invention Hold in the proof-pressure container filled up with inert gas, and to the heated crucible Raw material melt, In the manufacture approach of the compound semiconductor single crystal by the LEC method into which seed crystal and a crucible are rotated relatively and a single crystal is grown up, containing liquid encapsulant and contacting seed crystal to raw material melt The ratio {(crucible diameter) /(crystal diameter)} of the diameter of a crystal and a crucible to grow up grows up a compound semiconductor single crystal using the crucible set to the range of 2.2-3.2 (claim 3).
[0014] In the manufacturing installation or the manufacture approach of this invention, the diameter crystal of macrostomia 100mm or more can be dealt with as a diameter of the crystal to grow up (claims 2 and 4).
[0015] In an LEC method, the main point of this invention is making the ratio {(crucible diameter) /(crystal diameter)} of the diameter of a crystal and a crucible to grow up into the range of 2.2-3.2, and its repeatability is good and it makes it possible to obtain a compound semiconductor single crystal by high yield. In addition, as the diameter crystal of macrostomia 100mm or more, by making the ratio {(crucible diameter) /(crystal diameter)} of the diameter of a crystal and a crucible to grow up into the range of 2.2-3.2, the repeatability of the diameter of the crystal to grow up is more good, and it can obtain a compound semiconductor single crystal at high yield.
[0016] When the ratio of the diameter of a crystal and a crucible to grow up was less than 2.2, the minimum of the ratio of the diameter of the above-mentioned crystal which carries out growth, and a crucible was made or more into 2.2, because yield also became low, though the repeatability which lineage (Lineage) and a subgrain boundary are easy to be formed, and obtains a single crystal is low and natural. As a cause that lineage and a subgrain boundary are easy to be formed, it is presumed as follows. That is, although a solid-liquid interface must always be made into a convex at a melt side in order to obtain a single crystal, when the ratio of the diameter of a crystal and a crucible to grow up is 2.2 or less, a solid-liquid interface is not stabilized, but it is easy to generate the part which serves as concave at a melt side, and it is thought that a rearrangement concentrates and it leads to formation of lineage and a subgrain boundary.
[0017] Moreover, yield also becomes low, though having made or less into 3.2 the upper limit of the ratio of the diameter of the above-mentioned crystal which carries out growth, and a crucible has sharp fluctuation of the path of a crystal grown up when the ratio of the diameter of a crystal and a crucible to grow up exceeds 3.2, and its repeatability which obtains a single crystal is low and it is natural. As a cause by which fluctuation of the path of a crystal becomes intense, it is presumed as follows. That is, as for control of the diameter of a crystal in an LEC method, it is common to measure the weight of the crystal which grew up into per unit time amount, to compute the diameter of a crystal by processing this arithmetically, and to control the output of the heater which is a heating means. Since the responsibility to the path to control of the output of a heater becomes late when the ratio of the path of a crystal and the diameter of a crucible to grow up becomes large too much, it is thought that fluctuation of the path of a crystal to grow up becomes intense. In addition, if fluctuation of the diameter of a crystal is sharp, a solid-liquid interface will not be stabilized, either, but it will be easy to generate the part which serves as concave at a melt side, a rearrangement will concentrate, and it will lead to formation of lineage and a subgrain boundary.
[0018] In addition, though the crystal diameter to grow up is natural as the diameter crystal of macrostomia 100mm or more, as for the importance of a solid-liquid interface, it is stably needed from a small aperture crystal for a melt side increase and that it is a convex.
[0019]
[Embodiment of the Invention] Hereafter, the operation gestalt of this invention is explained focusing on an example. Here, the example applied to manufacture of the gallium arsenide which is a kind of a compound semiconductor is explained. In addition, the manufacturing installation used in the following examples 1-4 and examples 1-4 of a comparison contained raw material melt and liquid encapsulant to the crucible 5 which is the

same as that of drawing 1 , was held in the proof-pressure container slack elevated-temperature furnace 1 filled up with inert gas, and was heated, it rotated relatively the end crater 5 taken seed crystal 11, contacting seed crystal 11 to raw material melt, and manufactured the compound semiconductor single crystal by the LEC method. And especially in the examples 1-4, ratio K[of the diameter of the crystal to grow up and a crucible] = {(crucible diameter D) /(crystal diameter d)} used the crucible of the range of 2.2-3.2.
[0020] The contents of these examples 1-4 and the examples 1-4 of a comparison are shown in Table 1.
[0021]
[Table 1]

	るつぼ直径 [D]	結晶直径 [d]	結晶の長さ	るつぼ直径／結晶直径 [K]	全域単結晶
実施例 1	280	100	400	2.8	95%以上
実施例 2	220~320	100	400	2.2~3.2	90%以上
実施例 3	400	150	300	2.6	80%以上
実施例 4	330~480	150	300	2.2~3.2	75%以上
比較例 1	D<220	100	400	K<2.2	70%以下
比較例 2	D<330	150	300	K<2.2	60%以下
比較例 3	320<D	100	400	K<3.2	75%未満
比較例 4	480<D	150	300	K<3.2	65%以下

[0022] Single crystal growth of gallium arsenide with a crystal diameter [of 100mm] and a crystal die length of 400mm was performed 50 times using the crucible made from PBN whose [examples 1-2] diameter is 280mm (example 1). Consequently, the thing of a single crystal (All Single) all over the districts was obtained from seed attachment of a crystal by 95% or more of probability to the crystal growth last section.
[0023] Moreover, single crystal growth of gallium arsenide with a crystal diameter [of 100mm] and a crystal die length of 400mm was similarly performed 200 times using the crucible of the range whose diameter of the crucible made from PBN is 220mm - 320mm (example 2). Consequently, the thing of a single crystal all over the districts was obtained by 90% or more of probability.
[0024] [Examples 3-4] Single crystal growth of gallium arsenide with a crystal diameter [of 150mm] and a crystal die length of 300mm was performed 20 times again using the crucible made from PBN whose diameter is 400mm (example 3). Consequently, the thing of a single crystal all over the districts was obtained by 80% or more of probability.
[0025] Moreover, single crystal growth of gallium arsenide with a crystal diameter [of 150mm] and a crystal die length of 300mm was similarly performed 50 times using the crucible of the range whose diameter of the crucible made from PBN is 330mm - 480mm (example 4). Consequently, the thing of a single crystal all over the districts was obtained by 75% or more of probability.
[0026] As a result of gallium arsenide's with a crystal diameter [of 100mm] and a crystal die length of 400mm carrying out ******** length using the crucible made from PBN with the [examples 1-2 of comparison] diameter smaller than 220mm, the probability for the thing of a single crystal all over the districts to be obtained according to generating of lineage and a subgrain boundary etc. was 70% or less (example 1 of a comparison). In addition, the probability for the thing of a single crystal all over the districts to be obtained suited the inclination which becomes so low that the diameter of a crucible becomes small.
[0027] Moreover, as a result of performing single crystal growth of gallium arsenide with a crystal diameter [of

150mm], and a crystal die length of 300mm using the crucible made from PBN with a diameter smaller than 330mm, the probability for the thing of a single crystal all over the districts to be obtained according to generating of lineage and a subgrain boundary etc. was 60% or less like the case with a crystal diameter of 100mm (example 2 of a comparison).

[0028] The probability for fluctuation of the diameter of a crystal to be sharp, and to be in the middle of growth, for lineage and a subgrain boundary to occur as a result of performing single crystal growth of gallium arsenide with a crystal diameter [of 100mm] and a crystal die length of 400mm using the crucible made from PBN with the larger [examples 3-4 of comparison] diameter than 320mm, and for the thing of a single crystal all over the districts to be obtained was less than 75% (example 3 of a comparison). In addition, the probability for the thing of this single crystal all over the districts to be obtained suited the inclination which becomes so low that the diameter of a crucible becomes large.

[0029] Moreover, the probability for a case with a crystal diameter of 100mm and fluctuation of the diameter of a crystal to be sharp, and to be in the middle of growth, for lineage and a subgrain boundary to occur as a result of performing single crystal growth of gallium arsenide with a crystal diameter [of 150mm] and a crystal die length of 300mm using the crucible made from PBN with a larger diameter than 480mm, and for the thing of a single crystal all over the districts to be obtained was 65% or less (example 4 of a comparison).

[0030] Putting the above result together, repeatability is good and it turns out that it becomes possible to obtain a compound semiconductor single crystal at high yield because ratio K[of the diameter of the crystal to grow up and a crucible] = {(crucible diameter D) /(crystal diameter d)} chooses the diameter of a crystal to grow up, and the diameter of a crucible in 2.2-3.2. In addition, the diameter d of the crystal to grow up can obtain a compound semiconductor single crystal with more sufficient repeatability by high yield by making ratio K[of the diameter of the crystal to grow up and a crucible] = {(crucible diameter D) /(crystal diameter d)} into the range of 2.2-3.2 as the diameter crystal of macrostomia 100mm or more.

[0031] Although the above-mentioned example indicated the case where a gallium arsenide (GaAs) single crystal was grown up, this invention can be applied also about the manufacturing installation and the manufacture approach of a compound semiconductor single crystal of performing crystal growth by LEC methods, such as InP, GaP, and InAs, and can acquire the same effectiveness.

[0032] Moreover, the probability for the thing of a single crystal all over the districts to be obtained is not only high, but the compound semiconductor single crystal obtained with the approach by this invention and equipment has it in an inclination with little pile-up-of-dislocations section compared with the compound semiconductor single crystal obtained with the conventional method rather than a conventional method. Even if it is a single crystal all over the districts in the case of a conventional method, although this does not develop into lineage and a subgrain boundary, it shows that the rearrangement is being accumulated. The compound semiconductor wafer obtained by this invention can prevent the fall of the component yield based on a rearrangement, when a component is formed using this. A great thing has the economical effectiveness in industrial production.

[0033]

[Effect of the Invention] Since the diameter of a crystal and the diameter of a crucible into which the ratio {(crucible diameter) /(crystal diameter)} of the diameter of a crystal and a crucible to grow up is grown up so that it may go into the range of 2.2-3.2 are chosen according to this invention as explained above, a compound semiconductor single crystal can be obtained with sufficient repeatability by high yield using an LEC method. Therefore, the long crystal of a compound semiconductor single crystal and the diameter crystal of macrostomia can be obtained with sufficient repeatability.

[Translation done.]

* NOTICES *

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is drawing having shown the basic configuration of the manufacturing installation of the compound semiconductor single crystal by this invention.
[Description of Notations]
1 Elevated-Temperature Furnace
5 Crucible made from PBN
6 Ga,As
7 Liquid Encapsulant
8 Heater
11 Seed Crystal

[Translation done.]

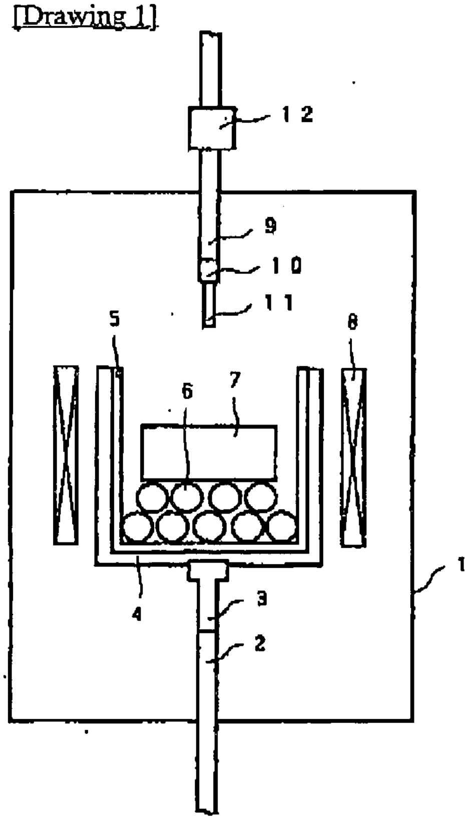
* NOTICES *

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DRAWINGS

[Drawing 1]



[Translation done.]

JUN. 25. 2007 6:13PM LNAPW 512-439-7199 NO. 1870 P. 27

(19)日本国特許庁（ＪＰ）

(12) 公開特許公報（Ａ）

(11)特許出願公開番号
特開2001－80989
（P2001－80989A）

(43)公開日 平成13年3月27日(2001.3.27)

(51)Int.Cl.7		識別記号	FI			テーマコード（参考）
C30B	15/10		C30B	15/10		4G077
	29/40	501		29/40	501A	5F053
	29/42			29/42		
H01L	21/208		H01L	21/208	P	

審査請求 未請求 請求項の数4 OL （全 6 頁）

(21)出願番号 特願平11－250244

(22)出願日 平成11年9月3日(1999.9.3)

(71)出願人 000005120
日立電線株式会社
東京都千代田区大手町一丁目6番1号

(72)発明者 和地 三千則
茨城県日立市日高町5丁目1番1号 日立電線株式会社日高工場内

(72)発明者 矢吹 伸司
茨城県日立市日高町5丁目1番1号 日立電線株式会社日高工場内

(72)発明者 水庭 清治
茨城県日立市日高町5丁目1番1号 日立電線株式会社日高工場内

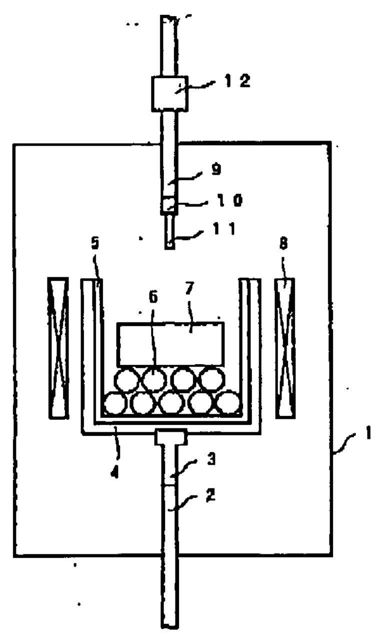
最終頁に続く

(54)【発明の名称】 化合物半導体単結晶の製造装置及びそれを用いた製造方法

(57)【要約】

【課題】化合物半導体単結晶の長尺結晶、大口径結晶を再現性良く得ることを可能にする。

【解決手段】不活性ガスを充填した耐圧容器内に収容され加熱されたるつぼ５に原料融液、液体封止剤を収納し、種結晶１１を原料融液に接触させつつ種結晶１１とるつぼ５とを相対的に回転させて、ＬＥＣ法により化合物半導体単結晶を製造するに際し、成長させる結晶とるつぼの直径の比｛（るつぼ直径）／（結晶直径）｝が２．２～３．２の範囲のるつぼを用いる。



4/13/07 EAST Version: 2.0.3.0
PAGE 27/30 * RCVD AT 6/25/2007 6:57:51 PM [Eastern Daylight Time] * SVR:USPTO-EFXRF-2/20 * DNIS:2738300 * CSID:512 327 5452 * DURATION (mm-ss):15-32

【特許請求の範囲】

【請求項１】不活性ガスを充填した耐圧容器内に収容され加熱されたるつぼに、原料融液、液体封止剤を収納し、種結晶を原料融液に接触させつつ種結晶とるつぼとを相対的に回転させて単結晶を成長させるＬＥＣ法による化合物半導体単結晶の製造装置において、成長させる結晶とるつぼの直径の比｛（るつぼ直径）／（結晶直径）｝を２．２〜３．２の範囲に定めたことを特徴とする化合物半導体単結晶の製造装置。

【請求項２】請求項１の化合物半導体単結晶の製造装置において、成長させる結晶の直径が１００mm以上の大口径結晶であることを特徴とする化合物半導体単結晶の製造装置。

【請求項３】不活性ガスを充填した耐圧容器内に収容され加熱されたるつぼに、原料融液、液体封止剤を収納し、種結晶を原料融液に接触させつつ種結晶とるつぼとを相対的に回転させて単結晶を成長させるＬＥＣ法による化合物半導体単結晶の製造方法において、成長させる結晶とるつぼの直径の比｛（るつぼ直径）／（結晶直径）｝を２．２〜３．２の範囲に定めたるつぼを使用して化合物半導体単結晶を成長させることを特徴とする化合物半導体単結晶の製造方法。

【請求項４】請求項２の化合物半導体単結晶の製造方法において、直径が１００mm以上の大口径の結晶を成長させることを特徴とする化合物半導体単結晶の製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、化合物半導体単結晶の製造装置及びそれを用いた製造方法に関するものである。

【０００２】

【従来の技術】III −V族化合物半導体はその単結晶の高品質化により、高速集積回路、光−電子集積回路やその他の電子素子に広く用いられるようになってきた。なかでも、砒化ガリウム（ＧａＡｓ）は電子移動度がシリコンに比べて早く、10^7 Ω・cm以上の比抵抗のウエハが製造容易という特徴がある。現在では上記半絶縁性ＧａＡｓの単結晶は、主に液体封止引き上げ法（ＬＥＣ法）により製造されている。

【０００３】図１に、ＧａＡｓ単結晶の製造装置として広く使用されているＬＥＣ法による結晶引上装置の概略を示す。

【０００４】図において、１は結晶成長用の高温炉（耐圧容器）であり、高温炉１内には下側から下軸２が挿入され、この下軸２の先端にペデスタル３を介してサセプタ４が支持されている。サセプタ４内にはＰＢＮ製るつぼ５が配置されている。サセプタ４の周囲にはヒータ８が設けられており、サセプタ４を介してＰＢＮ製るつぼ５を周囲から加熱できるようになっている。下軸２は図示しない回転機構に接続されており、一定の回転速度で回転されるようになっている。また、高温炉１の上側からは下軸２と同軸的に上軸９が挿入され、その下端に設けられた種結晶ホルダ１０に所望の方位を持った種結晶１１（通常、方位として（１００）が用いられる）が取り付けられる。この上軸９は、図示しない回転・昇降機構によってＰＢＮ製るつぼ５とは逆向きに軸回転されると共に、昇降移動されるようになっている。上軸９の途中には重量センサ１２が設けられており、これによって成長過程の結晶重量を検知できるようになっている。

【０００５】結晶成長の際には、先ずＰＢＮ製るつぼ５の中にＧａ，Ａｓ６（Ｇａ７４７０ｇ，Ａｓ８１２４ｇ）と、B_2O_3 液体封止剤７を４，０００ｇ入れ、高温炉１内を真空排気し、その後窒素またはアルゴンなどの不活性ガスで４０気圧程度に加圧し、主ヒータ８に通電してＰＢＮ製るつぼ５の内部を昇温させる。５００℃前後で液体封止剤（B_2O_3）７が軟化、融解して、Ｇａ，Ａｓ６を覆う。引き続き昇温させ、ＰＢＮ製るつぼ５内部の温度を１，２３８℃以上とし、ＧａＡｓを合成し、更に融解させる。次に、高温炉１内を５〜２０気圧に減圧した後、種結晶１１を降下させ、その先端を原料融液に浸して種付けを行う。その後、ヒータ８の温度を下げながら、上軸９を７〜１２mm/hrの速度で引き上げていき、重量センサ１２で結晶重量を検知しながら、ヒータ８の出力を制御してＧａＡｓ単結晶を成長させる。

【０００６】上記ＬＥＣ法で用いられるるつぼ５は、ＰＢＮ（Pyrolitic Boron Nitride ：熱分解窒化ホウ素）製を用いており、ＰＢＮ製るつぼは非常に高価である。このことから、ＬＥＣ法で用いられるるつぼの直径は、成長させる結晶の直径より若干大きなものを用いるのが一般的である。

【０００７】

【発明が解決しようとする課題】しかしながら、化合物半導体単結晶を成長する技術は非常に難しく、加熱手段のヒータ及び熱遮蔽筒等の部材（ホットゾーンと呼ばれる。以下ＨＺと記す）の配置、形状、材質等により、より細かく影響を受けるため、再現性の良い化合物半導体単結晶の成長条件を得るのは難しい。

【０００８】特に化合物半導体単結晶の直径が１００mmを超える大口径結晶を得ようとする場合等は、特に再現性の良い化合物半導体単結晶の成長条件を得ることは非常に難しく、ほとんどの場合は単結晶成長の過程において多結晶化してしまい、結晶の種付け部（シード部）から結晶成長最終部（テール部）まで、全域単結晶が得られることは希であった。

【０００９】図１には図示されていないが、高温炉１には熱遮蔽筒等の部材（ホットゾーンＨＺ）が設けられており、最近まで、このＨＺの配置、形状、材質等が化合物半導体単結晶の成長条件の再現性を左右する要因とみられていた。ところが、ＨＺの配置、形状、材質等の条件の安定化が図られるにつれ、その要因がＨＺのみにあ

晶とるつぼの直径の比K＝｛（るつぼ直径D）／（結晶直径d）｝が2．2〜3．2の範囲のるつぼを用いた。

【0020】この実施例1〜4及び比較例1〜4の内容＊を表1に示す。

【0021】

【表1】

	るつぼ直径 [D]	結晶直径 [d]	結晶の長さ	るつぼ直径／結晶直径 [K]	全域単結晶
実施例 1	280	100	400	2.8	95%以上
実施例 2	220〜320	100	400	2.2〜3.2	90%以上
実施例 3	400	150	300	2.6	80%以上
実施例 4	330〜480	150	300	2.2〜3.2	75%以上
比較例 1	D＜220	100	400	K＜2.2	70%以下
比較例 2	D＜330	150	300	K＜2.2	60%以下
比較例 3	320＜D	100	400	K＜3.2	75%未満
比較例 4	480＜D	150	300	K＜3.2	65%以下

【0022】［実施例1〜2］直径が280㎜であるPBN製のるつぼを用い、結晶直径100㎜、結晶長さ400㎜の砒化ガリウムの単結晶成長を50回行った（実施例1）。その結果、結晶の種付けから結晶成長最終部まで全域単結晶（All Single）のものが95%以上の確率で得られた。

【0023】また、PBN製るつぼの直径が220㎜〜320㎜の範囲のるつぼを用いて、同様に結晶直径100㎜、結晶長さ400㎜の砒化ガリウムの単結晶成長を200回行った（実施例2）。その結果、全域単結晶のものが90%以上の確率で得られた。

【0024】［実施例3〜4］また、直径が400㎜であるPBN製るつぼを用い、結晶直径150㎜、結晶長さ300㎜の砒化ガリウムの単結晶成長を20回行った（実施例3）。その結果、全域単結晶のものが80%以上の確率で得られた。

【0025】また、PBN製るつぼの直径が330㎜〜480㎜の範囲のるつぼを用いて、同様に結晶直径150㎜、結晶長さ300㎜の砒化ガリウムの単結晶成長を50回行った（実施例4）。その結果、全域単結晶のものが75%以上の確率で得られた。

【0026】［比較例1〜2］直径が220㎜より小さいPBN製のるつぼを用い、結晶直径100㎜、結晶長さ400㎜の砒化ガリウムの単結晶成長行った結果、リネージ、亜粒界の発生などにより、全域単結晶のものが得られる確率は70%以下であった（比較例1）。なお、全域単結晶のものが得られる確率は、るつぼの直径が小さくなる程低くなる傾向にあった。

【0027】また、直径が330㎜より小さいPBN製るつぼを用い、結晶直径150㎜、結晶長さ300㎜の砒化ガリウムの単結晶成長を行った結果、結晶直径10※0㎜の場合と同様にリネージ、亜粒界の発生などにより、全域単結晶のものが得られる確率は60%以下であった（比較例2）。

【0028】［比較例3〜4］直径が320㎜より大きいPBN製のるつぼを用い、結晶直径100㎜、結晶長さ400㎜の砒化ガリウムの単結晶成長を行った結果、結晶径の変動が激しく、成長途中でリネージ、亜粒界が発生し、全域単結晶のものが得られる確率は75%未満であった（比較例3）。なお、この全域単結晶のものが得られる確率はるつぼの直径が大きくなる程低くなる傾向にあった。

【0029】また、直径が480㎜より大きいPBN製るつぼを用い、結晶直径150㎜、結晶長さ300㎜の砒化ガリウムの単結晶成長を行った結果、結晶直径100㎜の場合と結晶径の変動が激しく、成長途中でリネージ、亜粒界が発生し、全域単結晶のものが得られる確率は65%以下であった（比較例4）。

【0030】以上の結果を総合すると、成長させる結晶とるつぼの直径の比K＝｛（るつぼ直径D）／（結晶直径d）｝が2．2〜3．2の範囲で、成長させる結晶径、るつぼ径を選択することで、再現性よく、且つ高収率で化合物半導体単結晶を得ることが可能になることが判る。なお、成長させる結晶の直径dが100㎜以上の大口径結晶では、成長させる結晶とるつぼの直径の比K＝｛（るつぼ直径D）／（結晶直径d）｝を2．2〜3．2の範囲とすることで、より再現性良く且つ高収率で化合物半導体単結晶を得ることができる。

【0031】上記実施例は、砒化ガリウム（GaAs）単結晶を成長させる場合について記載したが、本発明はInP、GaP、InAs等のLEC法で結晶成長を行う化合物半導体単結晶の製造装置及び製造方法について

PAGE 29/30 * RCVD AT 6/25/2007 6:57:51 PM [Eastern Daylight Time] * SVR:USPTO-EFXRF-2/20 * DNIS:2738300 * CSID:512 327 5452 * DURATION (mm-ss):15-32

も適用することができ、同様の効果を得ることができる。

【0032】また、本発明による方法、装置で得られる化合物半導体単結晶は、従来法よりも全域単結晶のものが得られる確率が高いだけではなく、従来法で得られた化合物半導体単結晶に比べ、転位の集積部が少ない傾向にある。これは、従来法の場合は、全域単結晶であっても、リネージ、亜粒界には発展しないまでも転位が集積していることを示している。本発明で得られる化合物半導体ウェハは、これを用いて素子を形成した場合、転位に基づく素子歩留の低下を防止できる。工業生産における経済的効果は多大なものがある。

【0033】

【発明の効果】以上説明したように本発明によれば、成長させる結晶とるつぼの直径の比｛（るつぼ直径）／（結晶直径）｝を２．２～３．２の範囲に入るように、

成長させる結晶径とるつぼ径を選択しているので、再現性よく且つ高収率で、ＬＥＣ法を用いて化合物半導体単結晶を得ることができる。従って、化合物半導体単結晶の長尺結晶、大口径結晶を再現性良く得ることができる。

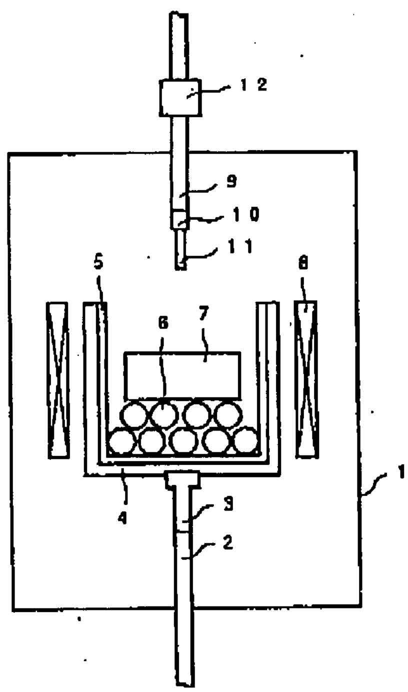
【図面の簡単な説明】

【図１】本発明による化合物半導体単結晶の製造装置の基本構成を示した図である。

【符号の説明】

１　高温炉
５　ＰＢＮ製るつぼ
６　Ｇａ，Ａｓ
７　液体封止剤
８　ヒータ
１１　種結晶

【図１】



フロントページの続き

(72)発明者　大宝　幸司
茨城県日立市日高町５丁目１番１号　日立電線株式会社日高工場内

(72)発明者　稲田　知己
茨城県日立市日高町５丁目１番１号　日立電線株式会社日高工場内

4/12/07 EAST Version: 2.0.3.0
PAGE 30/30 * RCVD AT 6/25/2007 6:57:51 PM [Eastern Daylight Time] * SVR:USPTO-EFXRF-2/20 * DNIS:2738300 * CSID:512 327 5452 * DURATION (mm-ss):15-32